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The effect of ion irradiation (Ar^+ , $E = 10$ keV) on the nanocrystalline structure and the aging of the Al-Li-Cu-Zn-Mg-Zr-Sc alloy after megaplastic deformation

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Abstract. The effect of Ar^+ ion irradiation on structural and phase transformations in a 1461 alloy (Al–Li–Cu–Mg) subjected to megaplastic deformation has been studied by transmission electron microscopy. Short-term irradiation ($E = 10$ keV, $F = 2 \times 10^{16} \text{ cm}^{-2}$) has been established to form a low-energy recrystallized submicrocrystalline structure at a depth ($\sim 200 \text{ }\mu\text{m}$), significantly exceeding the projective ion ranges ($\sim 10 \text{ nm}$). The study confirms the important role of the radiation-dynamic effect during the ion irradiation of metastable media. The structural and phase transformations take place in the alloy at a depth of much higher than the projective ion ranges and at a higher rate compared with traditional thermal annealing.

1. Introduction

Irradiation of ultrafine-grained and nanostructured materials with 10–40 keV gas ion beams enhances their structure and properties [1–3]. Accelerated ion-beam modification of Mo samples after severe plastic deformation by high pressure torsion, as well as the modification of bands of amorphous and nanocrystalline magnetically soft materials supports these results.

It is of interest to study the ion bombardment effect on other materials to comprehensively analyze how to control nanocrystalline structure formation, in particular, the grain size and uniformity.

The effect of argon ion irradiation on structural and phase transformations in the 1461 alloy (Al–2.8Cu–1.8Li–0.66Zn–0.5Mg–0.08Zr–0.09Sc) subjected to megaplastic deformations has been studied in this work using transmission electron microscopy.

2. Experimental

The 1461 alloy samples 2 mm thick were deformed at room temperature and a pressure of 4 GPa in Bridgman anvils to 5 revolutions (angle of the anvil rotation $\varphi = 10\pi$ rad). The thickness of the samples after deformation was $\sim 400 \text{ }\mu\text{m}$. The samples, when moving under ion beam at a rate of 1 sm/s in an ILM-1 ion implanter equipped with a PULSAR-1M source [4], were irradiated by Ar^+ ions in a continuous mode ($E = 10$ keV, $j = 100 \text{ }\mu\text{A/cm}^2$, $F = 6.25 \cdot 10^{15}$ and $2 \cdot 10^{16} \text{ cm}^{-2}$). The



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temperature of the samples was controlled under irradiation. It did not exceed 180°C at a maximum fluence.

The structure and the phase composition in thin alloy foils were examined using a Tecnai 30G² electron microscope at the Electron Microscopy Center of Collaborative Access of the Institute of Metal Physics, UB RAS. The foils were made from the samples by thinning them electrolytically from both sides. Thus, the initial and irradiated structures were examined at the center of the samples from a distance of 200 μm from both surfaces.

3. Details of exposure, measurements and results

A structure, which is a mixture of nanocrystalline and nanofragmented ones, formed in the 1461 alloy after megaplastic deformation (MPD) at $P = 4$ GPa, $\varphi = 10 \pi$ rad (figure 1). First structure resulted from dynamic recrystallization [5] occupies almost the entire volume, whereas the second one, only some areas of the sample. The diameters of recrystallized nanograins and nanofragments are 20–50 nm. The nanograins have an equiaxed shape and either straight-line or convex–concave boundaries. There is a contrast in the shape of arc and loops near nanograins with convex–concave boundaries. Elastic stress fields caused by unbalanced surface tension are responsible for this contrast. A weakly-pronounced banding with a band width of 50–100 nm is observed in some regions of the sample. According to [7], it can be explained by an MPD-induced increase in the lattice curvature. T_2 (Al_3CuLi_5)-phase particles 5–10 nm in diameter, nucleated heterogeneously are observed at nanograin boundaries.

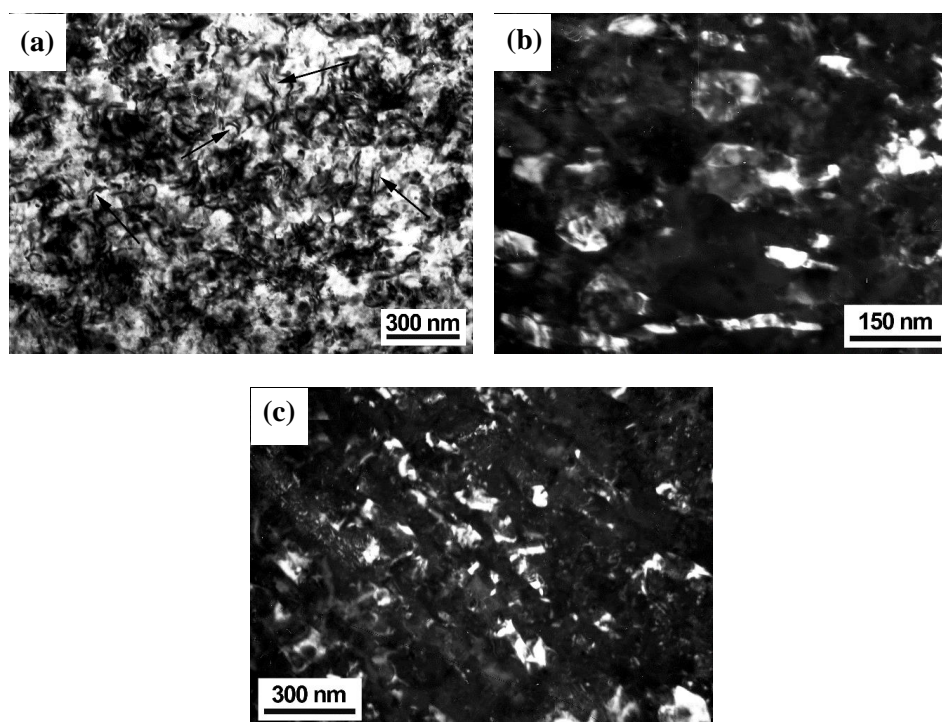


Figure 1. Microstructure of the 1461 alloy after MPD: (a) – bright-field image (dipole boundaries are shown by arrows) and (b), (c) dark-field images: (b) in reflection $(111)_{\text{Al}}$ and (c) in close reflections $(442)_{T_2} + (111)_{\text{Al}}$.

Electron-microscopy examination of the samples irradiated at a low ion fluence ($F = 6.25 \cdot 10^{15} \text{ cm}^{-2}$, irradiation time of 10 s) has showed that the irradiation initiates structure transformation in the severely deformed 1461 alloy and increases its structural nonuniformity. It manifests itself in the formation of coarse dislocation pileups (figure 2(a)).

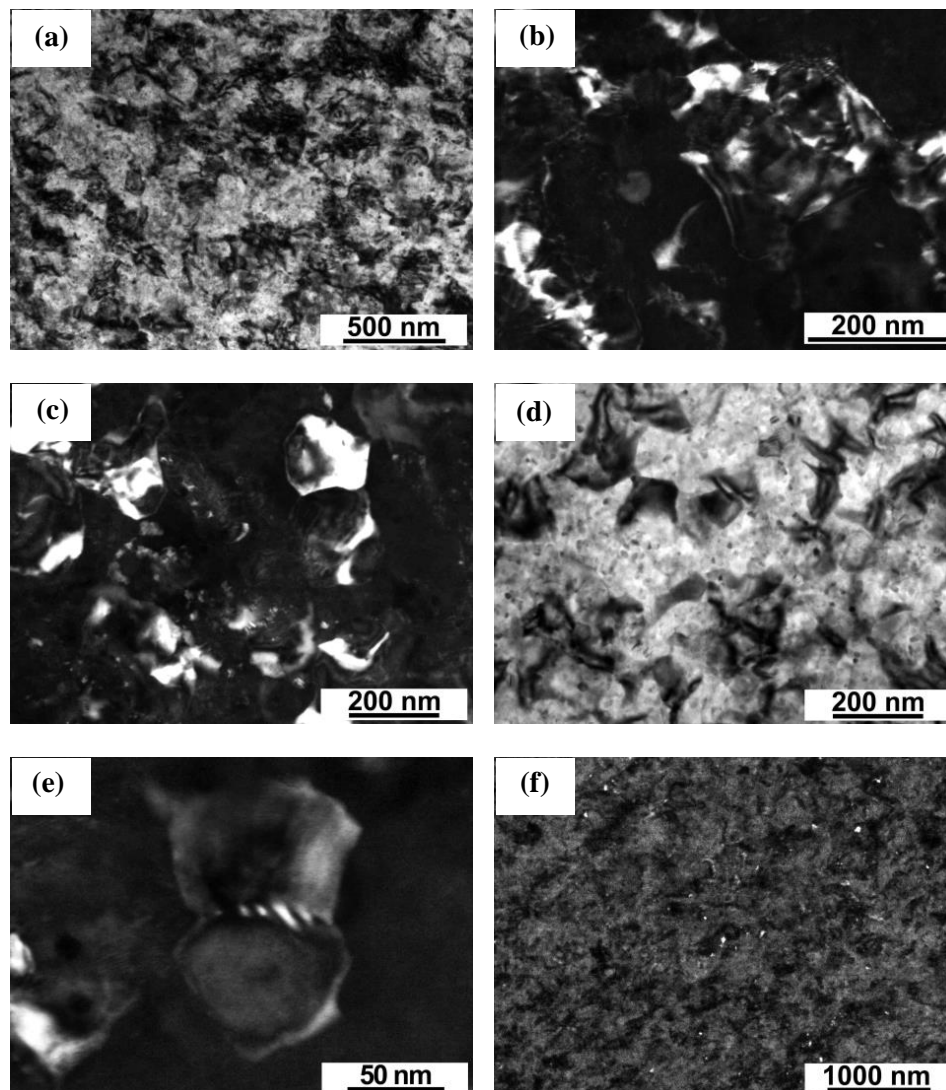


Figure 2. Microstructure of the 1461 alloy after MPD and Ar^+ ion irradiation ($E = 10$ keV, $j = 100 \mu\text{A}/\text{cm}^2$, $F = 6.25 \cdot 10^{15} \text{ cm}^{-2}$): (a), (d) bright-field images and (b), (c), (e), (f) dark-field images: (b), (c), (e) in reflection $(200)_{\text{Al}}$ and (f) $(530)_{\text{T}_2}$.

Moreover, the structure is nonuniform in grain sizes. The size of structural elements in regions with a high dislocation density varies within one dislocation tangle: there are nanograins 20–30 nm in diameter in the center and equiaxed submicrocrystals to 150 nm in diameter at the periphery (figure 2(b)). An almost uniform nano and submicrocrystalline structure with structural elements 100–120 nm in size is in regions with a low dislocation density (figures 2(c), (d)). Nanograins 50 nm and smaller in diameter have mainly nonequilibrium convex–concave boundaries, whereas those 70–100 nm in diameter and above have straight equilibrium boundaries (figure 2(e)). The irradiated alloy retains the elastically stressed state, which confirms the presence of fragments of dipole dislocations on its electron microscopic images (figure 2(d)). Such boundaries are known to be produced by MPD in Al–Li-based alloys and disappear during their subsequent recrystallization [6].

The irradiation in the considered mode does not have a noticeable effect on the supersaturated solid solution decomposition of a severely deformed alloy: there are heterogeneous nucleated T_2 -phase particles with a diameter to 10 nm at the boundaries of nano and submicrocrystals (figure 2(f)). Their sizes and distribution density are almost the same as those after MPD.

An increase in the ion fluence to $F=2 \cdot 10^{16} \text{ cm}^{-2}$ (irradiation time of 32 s) results in the formation of a uniform recrystallized submicrocrystalline structure with submicrocrystallite diameter of $\sim 200\text{--}400 \text{ nm}$ (figures 3(a), (b)). Submicrocrystallites have straight-line boundaries and equiaxial triple junctions (figure 3(c)). The electron microscopy images suggest the almost complete disappearance of dislocations. Dense dislocation tangles formed under lower fluence irradiation. This suggests that the formation of a recrystallized structure results from dislocation redistribution and subsequent annihilation, similar to that during annealing of deformed semi-finished products of coarse-grained materials. It should be noted that a low-temperature annealing (160°C , 15 h) forms a more dispersed recrystallized structure in the 1461 alloy deformed by MPD than the irradiation under considered conditions: diameters of nanograins and submicrocrystalline grains are $\sim 50\text{--}70$ and $\sim 150\text{--}170 \text{ nm}$, respectively [5].

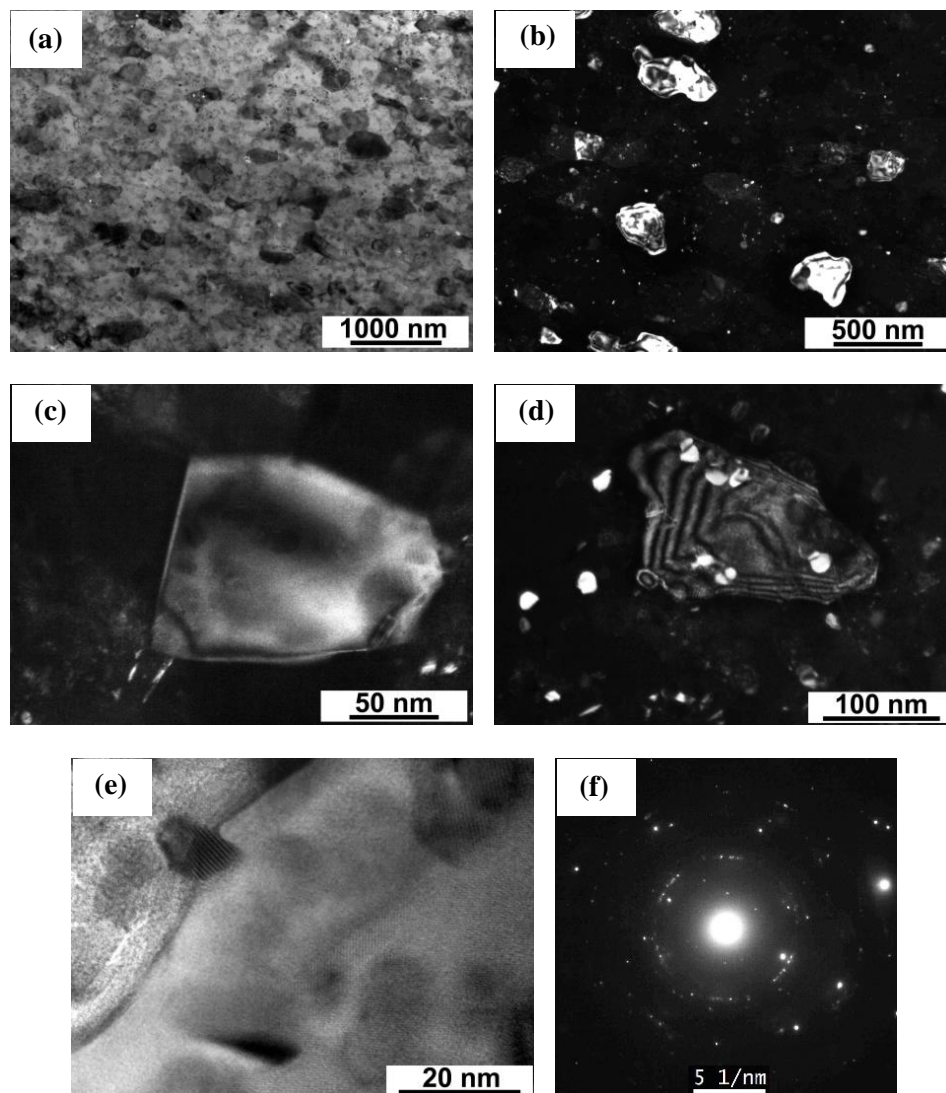


Figure 3. Microstructure of the 1461 alloy after MPD and Ar^+ ion irradiation ($E = 10 \text{ keV}$, $j = 100 \mu\text{A}/\text{cm}^2$, $F = 2 \cdot 10^{16} \text{ cm}^{-2}$): (a) – bright-field image; (b)–(d) – dark-field image in reflections $(200)_{\text{Al}} + (530)_{\text{T}_2}$, (e) – in reflection $(200)_{\text{Al}}$, and (f) – ring electron diffraction pattern.

Irradiation at a higher fluence activates supersaturated solid solution decomposition. Heterogeneous nucleation and growth of T_2 -phase particles are observed (figure 3(d)). Their average diameter after

irradiation is ~10–15 nm. Qualitative analysis also revealed an increase in their distribution density. In addition to the T_2 phase, there are ultradisperse metastable δ' -phase (Al_3Li) precipitates with an ordered structure of the $L1_2$ type in the irradiated alloy: bright-field and dark-field images show a weakly pronounced ripple contrast (figure 3(e)) and corresponding electron diffraction patterns show weakly pronounced superlattice reflections (figure 3(f)). The diameter of the particles is difficult to estimate due to their fine size.

Additionally, the irradiation under the considered conditions depletes the solid solution with alloying elements due to an increase in the volume fraction of the T_2 phase and δ' -phase precipitations. This can contribute to both an intensive redistribution of dislocations followed by annihilation and the growth of recrystallization nuclei, since the atoms of alloying elements in a solid solution prevent the redistribution of dislocations and, ultimately, the nucleation and the growth of recrystallized grains.

4. Conclusions

Transmission electron microscopy examination showed that short-term (10–32 s) Ar^+ ion irradiation at an ion energy of 10 keV significantly changes the structure of the deformed 1461 alloy. Structural changes depends on the fluence of Ar^+ ions. The irradiation of the alloy at a low fluence of $6.25 \cdot 10^{15} \text{ cm}^{-2}$ (10 s) initiates large dislocation pileups, increases the nonuniformity of the grain size, but does not cause relaxation of elastic stresses and does not affect the kinetics of nucleation and growth of the T_2 phase. An increase in the fluence to $2 \cdot 10^{16} \text{ cm}^{-2}$ (32 s irradiation) activates the precipitation of excess phases and the formation of a low-energy uniform recrystallized structure. Structural and phase transformations take place in the alloy at a depth (~200 μm) that significantly exceeds the projective ion range (~10 nm) at a higher rate as compared to traditional thermal annealing, which confirms the important role of the radiation-dynamic constituent under ion irradiation of metastable media [3].

Acknowledgments

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References

- [1] Ovchinnikov V V, Gushchina N V, Gapontseva T M, Chashchukhina T I, Voronova L M, Pilyugin V P and Degtyarev M V 2015 *High Pressure Research* **35** (3) 300–9
- [2] Ovchinnikov V V, Makhin'ko F F, Gushchina N V et al. 2017 *Physics of Metals and Metallography* **118** (2) 150–7
- [3] Ovchinnikov V V 2018 *Surface and Coating Technology* **355** 65–83
- [4] Gavrilov N V, Mesyats G A, Nikulin S P, Radkovskii G V, Eklind A and Perry A J 1996 *J. Vac. Sci. Technol. A* **14** 1050–6
- [5] Kaigorodova L I, Rasposienko D Y, Pushin V G, Pilyugin V P and Smirnov S V 2018 *Physics of Metals and Metallography* **119** (2) 161–8
- [6] Kaigorodova L I, Rasposienko D Y, Pushin V G, Pilyugin V P and Smirnov S V 2015 *Physics of Metals and Metallography* **116** (4) 346–55
- [7] Ditenberg I A, Tyumentsev A N, Grinyaev K V, Chernov V M et al. *Technical Physics. The Russian Journal of Applied Physics* **56** (6) 815–20